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PROVISIONAL APPLICATION FOR PATENT COVER SHEET

This is a request for filing a PROVISIONAL APPLICATION FOR PATENT under 37 CFR 1.53(b)(2)

Docket Number	UoA10	Type a plus sign (+) inside this box →	X

INVENTOR(s)/APPLICANT(s)

LAST NAME	FIRST NAME	MIDDLE INITIAL	RESIDENCE (CITY AND EITHER STATE OR FOREIGN COUNTRY)
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TITLE OF THE INVENTION (280 characters max)

Process for Treating Fine Coal Particles

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ENCLOSED APPLICATION PARTS (check all that apply)

☒ Specification combined	Number of Pages	18	x	Small Entity Statement
☒ Drawing(s)	Number of Sheets		☐	Other (specify)

METHOD OF PAYMENT OF FILING FEES FOR THIS PROVISIONAL APPLICATION FOR PATENT (check one)

☐	A check or money order is enclosed to cover the filing fees		FILING FEE AMOUNT(S)	75.00
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Respectfully submitted,

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TYPED OR PRINT NAME C.A.Rowley REGISTRATION NO. 20 781
(if appropriate)

☐ Additional inventors are being named on separately numbered sheets attached hereto.

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6 July 1999.

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Dear Sir: **RE: New Provisional Patent Application** **My file no. UoA10**
Title: Process for Treating Fine Coal Particles
Inventor(s): Jaewon Choung; Jozef Szymanski and Zhenghe

Please file the enclosed Provisional patent application, enclosed are

The patent application with drawings incorporated in the Specification and a cover sheet.

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Agent for Applicant
Registration No. 20,781

Process for Treating Fine Coal Particles

ABSTRACT

A process for recovering fine coals and clarifying water for recycling features with three distinct mechanisms: hydrophobic extraction, electrolyte coagulation, and macromolecular flocculation. If desired these three mechanisms may be integrated into a single stage. The hydrophobic extraction is accomplished using oil to form a coal-in-oil mixture. The process water is clarified using a flocculant, more preferably a combination of coagulant and anionic flocculant. With a well defined reagent addition scheme, this process allows a combustible recovery greater than 88% at an ash content less than 11%, while producing a clean water containing less than 20 ppm suspended solids from a tailing's stream. The coal-in-oil mixture may be used as a fuel for example in coal-firing boilers and power generators.

INTRODUCTION

A recognized challenge in coal industry is inefficient recovery of coal fines, compounded with the trend of producing more fines with continuous mining systems and subsequent coal preparation. Until very recent years, fine coals were merely washed to large volume of tailings stream, not only losing energy resources but also having some environmental consequences. A modern coal preparation plant at Smoky River Coal Ltd. (Granade Cache, Alberta), for example, produces more than 900 m^3/hr tailings containing less than 3% solids. About 60% of discarded fine solids is coal. Considering that such a large quantity of water has to be treated before recycle or safe discharge to the environment, economics dictate that as much of the marketable coal as possible should be recovered and water recycled if possible. From both economic and environmental considerations, the incentive to recover fine coal from a tailings stream becomes evident.

A few processes have been tested for recovering fine coals from tailings stream, including froth flotation and oil agglomeration. Although froth flotation featuring with microbubble technology had some success in recovering fine coals, thermal efficiency of the recovered coal is in general low due to the presence of surface moisture, encumbered by

large surface areas (Norton, 1984). In addition, the discharge from flotation circuit remains to contain large amount of mineral matters including fine clays, which are difficult to remove by conventional filtration or thickening. Oil agglomeration, on the other hand, appears to be promising in recovering fine coals, as there is virtually no lower particle size limit and the ultimate floatability of coal is less affected by size compared to froth flotation (Mehrotra, et al., 1983). An important feature of oil agglomeration process is low moisture content of the products. However, the recovered fine coals still need to be pelletized by adding an extra amount of heavy oil and screened to a few millimeters in diameter, to facilitate the storage and handling and to ensure an effective flame contact in combustion chambers. As in froth flotation, a second stage treatment to remove fine particles from process effluent for water recycle is needed with the oil agglomeration process.

Brief Description of the Invention

An object of the invention is to provide an economic process capable of recovering fine coals and cleaning up process water for recycle, preferably in an integrated, single stage operation. Broadly the present invention related to processing coal fines comprises mixing an organic liquid (oil) with the coal fines suspended in an aqueous phase, the oil being applied in an amount to extract the coal fines by hydrophobic extraction, thereby leaving hydrophilic mineral matters in the aqueous phase. Either a flocculating agent, a coagulating agent or a combination of the two is added to the aqueous phase to facilitate the separation of fine particles remaining in the aqueous phase, which allows recycling or safe dispose of the clarified aqueous phase. Preferably the mixing of the oil and a flocculating agent, a coagulating agent or a combination of the two for recovering fine coals and removing fine suspended solids from the aqueous phase to provide an aqueous phase for recycling is integrated into a single stage. The coal-in-oil mixture, clarified aqueous phase and the fine particles are separated in a modified thickener. Preferably the separation of fine suspended solids from the aqueous suspension is by using a combination of electrolyte coagulation and macromolecular flocculation.

Description of the invention

An aqueous suspension of fine coal particles containing other particles such as fine clay particles, as found for example in coal tailings, is mixed for a period of time with an oil (preferably a low grade fuel oil) and preferably at the same time with either a flocculating agent, a coagulating agent or a combination of the two. The coal fines are extracted into the oil phase which floats on the aqueous phase (floats - recoverable coal fines), and the other particles (rejects) sink, leaving a clarified aqueous phase (recycle water) there between. The oil phase containing the coal fines forms a coal-in-oil mixture that may be used directly as a fuel product.

Following the conditioning, the separation is carried out in the same vessel, or if desired the floats may be separated in one vessel and the other fine particles in a second vessel or pond to provide the clarified water. Generally the suspension will contain particles in the size range below 2mm. Generally the incoming aqueous suspension contains a solids content in the range of about 1 to 10 % by dry weight solids with the coal comprising about 20 to 70 % by weight of the solids. The extraction oil will be added in the amount of between about 100 and 250 % based on the dry weight of the coal fines in the suspension. Preferably the oil will be selected from the group including heavy crude, light mineral oils and fuel oils. The flocculating agent will normally be added in the concentration below 30 ppm, a coagulating agent in the concentration of up to about 1000 ppm, or a combination of the two in the ratio of between 1/10 and 1/100 of flocculant to coagulant. The preferred flocculant for use in the present invention includes cationic or anionic flocculants and the preferred coagulant includes positively charged aluminum hydrosols or calcium cations. When a combination of flocculant and coagulant is used, the preferred combination will contain an anionic flocculant and calcium cations.

Examples

Sample Preparation

The gravity circuit concentrate from Smoky River Coal Ltd. was used in this study. The coal chunks retained on 1½" screen after wet-screen were further cleaned using a heavy fluid of density 1.35 (mixture of Perchloroethylene and Varsol). The floats were thoroughly

washed with tap water and pulverized in a laboratory pulverizer (Brinkmann Instruments Ltd., Type ZM-1). The pulverized coal was sieved into various size fractions, each sealed in a glass bottle and stored in a freezer at -4 °C. The samples prepared as such were referred to as clean coal with ash content for each size fraction being given in Table 1. To control surface hydrophobicity, the clean coal sample was further oxidized in a well-ventilated oven at 150, 175 or 200 °C for 24 hours prior to extraction tests. The thin film flotation (Williams and Fuerstenau, 1987) was conducted to determine the wetting characteristics of the resultant samples, which was considered as a measure of surface hydrophobicity. In these tests, methanol-water mixtures of varying concentrations were used as probing liquids.

TABLE I - Ash Distribution in Pulverized Clean Coal Samples Used in the Case Studies.

Size (μm)	Weight (%)	Ash (%)
+ 1180	4.68	4.59
1180 x 589	11.15	4.43
589 x 417	9.35	4.51
417 x 208	25.61	4.40
208 x 150	16.91	3.96
150 x 74	13.31	4.23
74 x 38	16.55	4.30
-38	2.44	4.33
Total	100.00	4.31

A fine coal tailings sample taken from the tailings stream of the 2nd bank of flotation cells at Smoky River Coal Ltd. was tested. To simplify the test procedures, the solids in the stream were collected after filtration and dried at 105 °C. The fine solid samples with a top particle size of 500 μm contained 37.7% mineral matters. To study the effect of particle size

on process performance, a portion of the sample was dry-screened to obtain -45 μm size fractions. The sample obtained as such contained 41.1% mineral matters.

Chemicals

Analytical grade light mineral oil was purchased from Anachemia, Canada. The cationic (Magnifloc 591-C, MW of ~200,000), anionic (Superfloc 218 Plus, MW of ~10^6) and nonionic (Superfloc 127 Plus, MW of ~10^6) flocculants were provided generously by Cytec Industries Inc., USA. Analytical grade calcium chloride was purchased from Fisher Scientific, Canada. All the chemicals were used as received. Unless otherwise stated, all the experiments were carried out at room temperature with de-ionized water prepared using an Elix-5, followed by the purification with a Millipore-UV unit (Millipore, Canada).

Procedures

The batch type experiments were conducted in a 400 mL glass beaker. The coal slurry was prepared by adding 5.76 grams of dried sample into 200 mL of de-ionized water under mechanical agitation for 5 minutes at 1300 rpm (Caframo Lab. Stirrer 4405-10, Cole-Parmer). This resulted in a pulp of 2.8% solids, corresponding to the percent solids in the original coal tailings stream.

In a typical extraction test with clean coal samples, the desired amount of mineral oil, flocculant and/or coagulant were added into the resultant coal slurry under mechanical agitation. A conditioning period of 10, 2 and 0.5 minutes after the addition of mineral oil, coagulant and flocculant, respectively, was allowed, with the order of addition being altered. After conditioning, the slurry was poured as quickly as possible into a separatory cylinder. Following a 15-minute settling during which phase separation occurred, the rejects (aqueous phase) and floats (organic phase) were separated into a beaker and an aluminum pan, respectively. The solids retained in the aqueous phase were filtered on a filter circle fixed on a funnel, washed with toluene, dried in the oven at 105 °C for 5 hours, and weighed. The solids extracted in the oil phase were then determined from mass balance. The yield of solids retained in the oil phase was used to evaluate the extraction performance. Similar procedures were applied to the tailings sample. In this case, the ash content of the feed and solids in the aqueous phase was determined in accordance with ASTM (D 3174). The combustible

recovery and ash content in organic phase were then calculated based on the solid mass of the feed and rejects, along with their ash contents. The amount of suspended solids remaining in the treated water was determined by a spectrophotometer (Spectronic 21, Spectronic Instruments, Inc.) after calibration.

RESULTS/DISCUSSION

Thin film flotation

The results from thin film flotation tests are shown in Figure 1. It is clear that with increasing the oxidation temperature, coal surface gets progressively oxidized as indicated by a decreased fraction of solids in floats for a given probing liquid. The significant decrease, however, occurred at an oxidization temperature of 175 °C as the thin film flotation curve changed from convex to concave shape. A similar observation was reported earlier for other coal samples (Xu and Yoon, 1989).

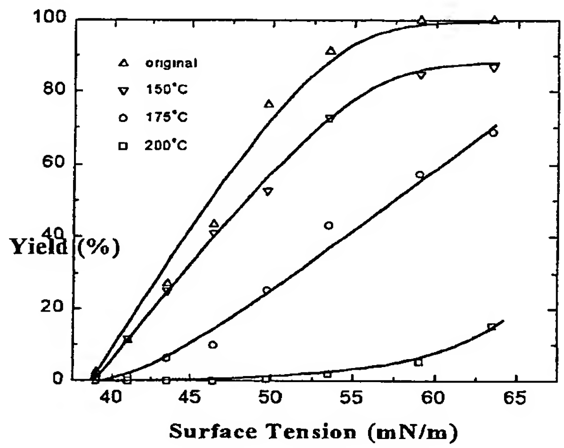


Figure 1. Effect of oxidation temperature on coal surface hydrophobicity evaluated by thin film flotation (coal particles in - 417 + 208 μm size fractions were oxidized for 24 hours).

Hydrophobic Extraction

Role of surface hydrophobicity: The role of surface hydrophobicity in hydrophobic extraction of coal into oil phase is shown in Figure 2. For clean coal samples without further

oxidation, obtained is an extraction yield of almost 100% coal in oil phase as either agglomerates (at oil/solid ratio below 1) or coal-in-oil mixture (at oil/solid ratio above 1). The results suggest the existence of a minimum oil to solid ratio of 1 for coal extraction in contrast to oil agglomeration. Also noted in Figure 2 is a negligible effect of coal oxidation at up to 150 °C, although a reduction in surface hydrophobicity was noted under these oxidation conditions (Figure 1). A significant decrease in coal extraction, in particular at oil to solid ratio below 1, was observed with coal samples oxidized at 175 °C. This transition corresponded well with that found in thin film flotation tests. Further increase in oxidation temperature caused a further decrease in extractability of coal into the oil phase. These findings illustrate the important role of surface hydrophobicity in coal extraction by oil. Clearly, a minimum surface hydrophobicity with a critical surface tension of ca. 55 mN/m is needed for effective extraction of coal by oil. Incidently, this surface tension value corresponded well with typical mineral oil-water interfacial tensions (Girifalco and Good, 1957).

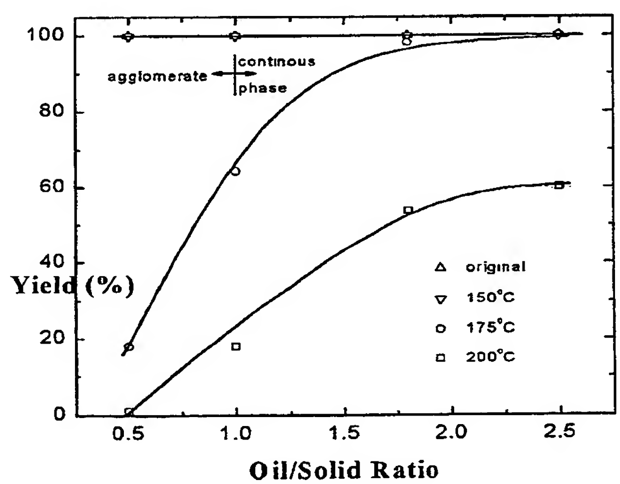


Figure 2. Effect of surface hydrophobicity on hydrophobic extraction as a function of oil/solid ratio (coal particles in - 417 + 208 μm size fractions were oxidized for 24 hours).

Effect of particle size: The effect of particle size on hydrophobic extraction of coal by mineral oil was examined at the oil to solid ratio of 1.8 to ensure a process of hydrophobic extraction as oppose to oil agglomeration. The results in Figure 3 show that with strongly hydrophobic (clean) coal samples, the extraction yield reached almost 100% for all the size fractions examined. This finding suggests that over a wide particle size range, the hydrophobic extraction is insensitive to particle sizes. The similar extraction results were obtained with coal samples oxidized at up to 175 °C. However, with coal samples oxidized at 200 °C, a significant decrease in extraction yield was observed with the samples in coarser size fractions (grater than 200 μm size). Clearly, the upper limit of sizes for particles to be extractable is determined by the balance of the gravitational force against interfacial tension force, which is a strong function of surface hydrophobicity.

Effect of additives: Since one of our objectives is to develop a single step process capable of recovering clean coals from coal tailings stream and at the same time clarifying the process water for recycle or safe disposal, it is important to examine the effect of flocculant or/and coagulant to be used in clarification on extractability of fine coals. As shown in Fig. 4, when added before the mineral oil (oil to solid ratio of 1.8), the cationic flocculant showed no effect on extractability of clean (fresh) coal samples with an almost 100% yield being retained. For coal samples oxidized at 150 °C, however, a decreased yield with increasing flocculant concentration was observed. This decrease in extraction yield continued with further increase in oxidation temperatures. It appears that the coal particles carry a net negative surface charge over the pH range studied (between 5 to 7) and the increased negative surface charge upon oxidation of coal enhanced adsorption of cationic flocculant, which further reduced the surface hydrophobicity. Another contributing factor for the decreased yield with increasing cationic flocculant concentration could be the increased aggregate size arising from flocculation of increasingly oxidized coal particles. With the anionic flocculant, however, the addition of flocculant did not show substantial effect on the extraction yields of coal samples oxidized at various temperatures, as seen in Figure 5. This finding further confirms that the coal surface carriers progressively more negative surface

charges upon oxidation, which did not respond to the addition of the anionic flocculant. The electrostatic repulsive force between anionic flocculant and negatively charged coal surface appears to prevent the flocculant from adsorbing on coal, showing no effect on surface hydrophobicity and hence extraction yield of coal samples. A noticeable decrease in extraction yield for coal samples oxidized at 200 °C may be an indication of marginal flocculant adsorption through hydrogen bonding, hindered by the electrostatic repulsive forces.

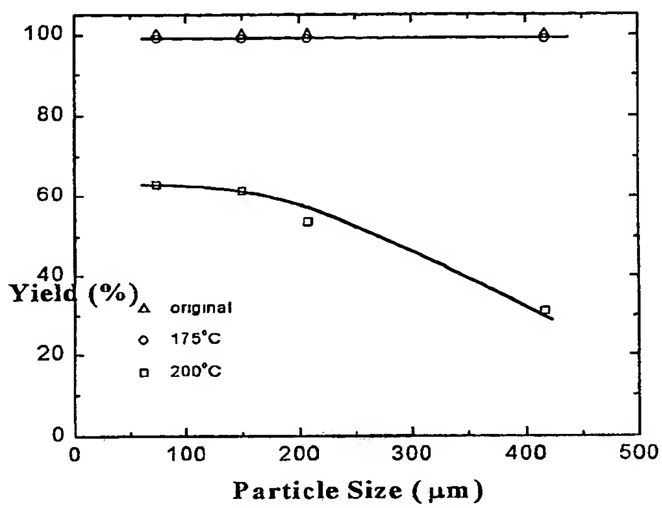


Figure 3. **Effect of particle size on hydrophobic extraction at oil/solid ratio of 1.8.**

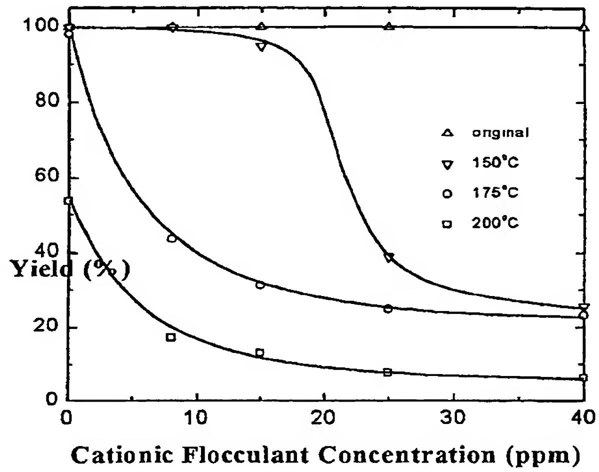


Figure 4. **Effect of cationic flocculant added prior to mineral oil on hydrophobic extraction at oil/solid ratio of 1.8 (coal particles in - 417 + 208 μm size fractions were oxidized for 24 hours).**

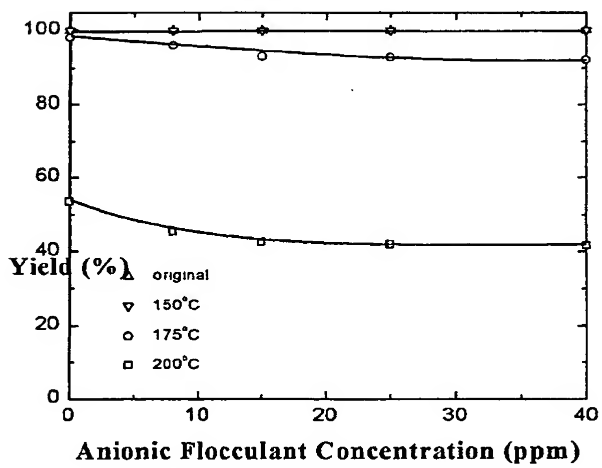


Figure 5. **Effect of anionic flocculant added prior to mineral oil on hydrophobic extraction at oil/solid ratio of 1.8 (coal particles in - 417 + 208 μm size fractions were oxidized for 24 hours).**

It was anticipated that the use of inorganic coagulants such as positively charged aluminum hydrosols or calcium cations, before the mineral oil addition, could decrease the extraction yield by decreasing hydrophobicity or/and increasing the size of coal aggregates, should coagulation occur. However, this effect was not observed with coal samples oxidized below 175 °C. A marginal decrease in coal extraction was observed with coal samples oxidized at temperatures higher than 175 °C, as shown in Figure 6 (open symbols). It appears that only at a sufficiently high oxidation temperature, can coal surface carbon become oxidized to carboxylic groups amenable for specific adsorption by calcium, thus reducing the hydrophobicity and hence hydrophobic extractability.

As shown in Figure 6 (solid symbols), a significant decrease in extraction yield was observed for coal samples oxidized at temperatures as low as 150 °C when anionic flocculant (25ppm) and calcium were present. It is clear that the adsorption of calcium in the electrical double layer appears to be sufficient to induce the adsorption of anionic flocculant, which decreases the surface hydrophobicity and increases aggregate size by flocculation as visually observed. As a result, a decreased extraction yield is anticipated as observed experimentally. This synergetic action by calcium and anionic flocculant was further enhanced with coal samples oxidized at higher temperature.

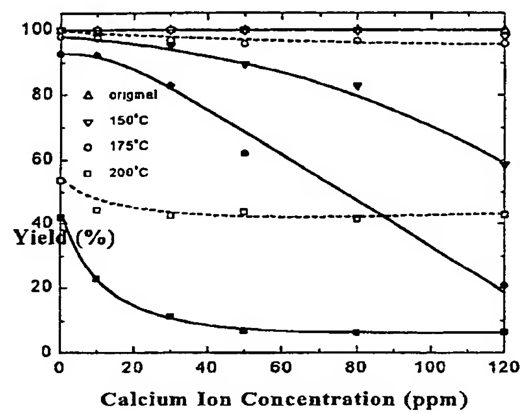


Figure 6. Effect of coagulant added prior to mineral oil on hydrophobic extraction at oil/solid ratio of 1.8 (coal particles, - 417 + 208 µm; solid symbols, with anionic flocculant; open symbols, no anionic flocculant).

The above results clearly show that for hydrophobic coals, it is feasible to recover them by hydrophobic extraction from a slurry in the presence of the coagulant and flocculant needed to aid the clarification of the effluent in a single step. For weakly hydrophobic coals, however, the presence of coagulant and flocculant hindered coal extraction into oil phase. In this case, it is vital to extract the coal into the oil phase prior to flocculant/coagulant addition to make the process work. To test this hypothesis, the extraction tests were conducted by adding the mineral oil to a coal slurry first. The results given in Table 2 show that adding coagulant and flocculant after the mineral oil did not change the extraction yield of coal oxidized at 175 and 200 °C. In contrast, a significant decrease in extraction yield from 98 and 55% to 62 and 7% was observed for coal samples oxidized at 175 and 200 °C, respectively, when the mineral oil was added after coagulant and flocculant. This finding confirms the importance of the chemical addition scheme. Clearly, after hydrophobic coal particles being extracted into the organic phase, water-soluble species has little effect on those particles in the organic phase, but would act on the particles in aqueous phase. It appears that with the well defined reagent addition scheme, the integrated approach would

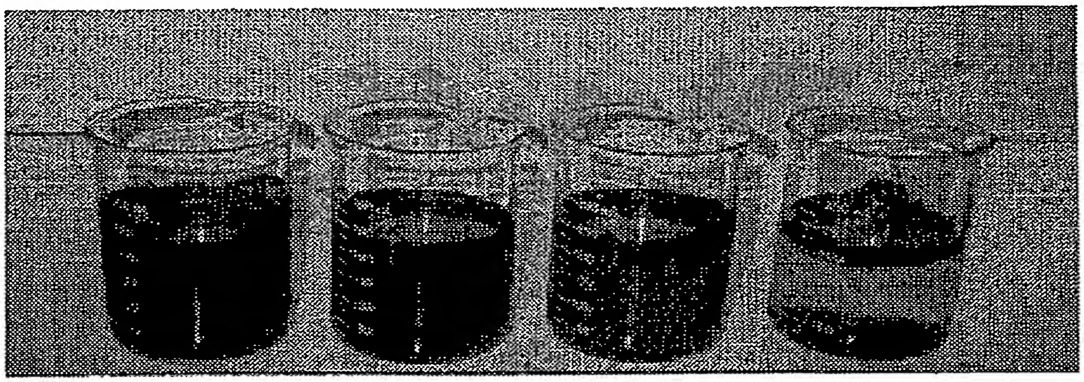
work satisfactorily even with less hydrophobic (oxidized) coals that froth flotation may not be effective to recover.

TABLE II - Effect of Reagent Addition Order on Process Performance.

Order of Reagent Addition	% Yield from Oxidized Coal	
	175 °C	200 °C
Oil Extraction Only	98	53
Oil Extraction Followed by Reagent Addition	98	55
Reagent Addition Followed by Oil Extraction	62	7

Integrated Process

The integration of three mechanisms, i.e. hydrophobic extraction of fine coals and a combination of coagulation and flocculation to clarify process water for recycle, in a single process is demonstrated in a case study with an industrial coal flotation tailings sample taken from Smoky River Coal Ltd. As shown in Figure 7a, adding clear mineral oil into the coal tailings slurry at oil to coal ratio of 1.8, resulted in a black, continuous oil phase on the top of aqueous phase, confirming hydrophobic extraction of fine coals in oil phase. Also shown in Figure 7a is a cloud aqueous phase (lower part) even after the suspension was left overnight, illustrating the need for clarification of the effluent for recycling. This features the second novelty of the process.



a) b) c) d)

Figure 7. **Photographic illustration of hydrophobic extraction, flocculation and/or coagulation in a single stage process: a) hydrophobic extraction, b) a plus electrolytic coagulation, c) a plus flocculation, and d) all three mechanism, i.e. a), b) and c).**

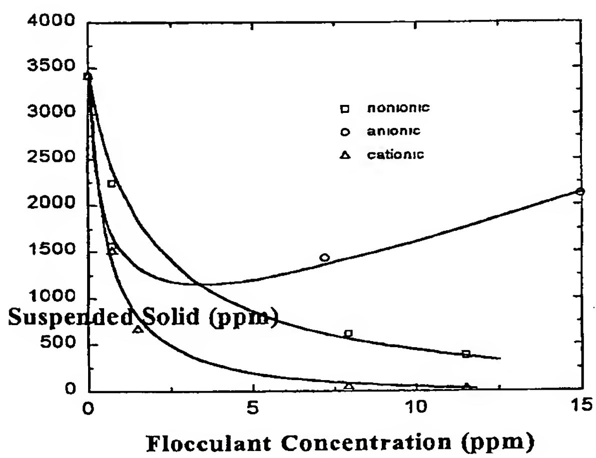


Figure 8. **Clarity of processed water as a function of flocculant concentration.**

As shown in Figure 8, the addition of cationic flocculent significantly reduced the amount of solids remaining in the processed water. However, the bulk aqueous phase remained dark as seen in Figure 7c. Apparently, this water cannot be discharged without further treatment. Compared to the cationic flocculent, the nonionic and anionic flocculants were found less effective as shown in Figure 8. Adding 100 ppm calcium alone as coagulant was able to clarify the effluent from an original solid content of 3500 ppm to 410 ppm, but the supernatant remained dark as seen in Figure 7b. These results suggest that flocculant or coagulant alone is unlikely to clarify the effluent to the desired level for safe discharge or recycle. A combination of coagulation and flocculation is therefore considered. A sharp decrease of suspended solids in the treated water was indeed observed when calcium and anionic flocculant were added together. In the presence of 1.5 ppm anionic flocculant, the addition of 100 ppm calcium resulted in a reduction in suspended solids from 1250 ppm to below 20 ppm, with a clear water zone sandwiched between a black top oil phase and bottom sediments as seen in Figure 7d. The quantitative analysis showed that this process is capable of achieving a combustible recovery greater than 88% at a product ash content less than 11%, while producing a clean water containing less than 20 ppm suspended solids (Table 3). The improved product ash content from 21.7 to 10.5% with decreasing feed particle size, is attributed to the increased degree of liberation with decreasing particle size. In both cases, the oil recovery in organic phase is close to 100% and the solid content in discharge water meets the limit (20 ppm) for recycle and discharge (Down, 1977).

TABLE III - Effect of Feed Particle Size on Process Performance

Analysis	-325 Mesh Fraction	Tailing as Received
Ash Content in Feed (%)	41.1	37.7
Ash Content in Float (%)	10.5	21.7
Ash Content in Reject (%)	81.7	80.0
Combustible Recovery (%)	88.8	91.2
Suspended Solid in Water (ppm)	18.0	15.0

It should be noted that the hydrophobic oil extraction featured in this process is conceptually different from the oil agglomeration process, in which case oil is used as a bridge to link particles. The current process produces a liquid fuel in the form of coal-in-oil mixture (COM). As a result, the moisture content entrapped by fine coal in oil phase is minimal compared to the moisture in flotation froth product even after dewatering. Therefore, a higher thermal efficiency is anticipated from coal-in-oil fuel. Another added advantage to produce liquid fuel from coal tailings is the easy handling and transportation of slurry product. The COM is currently used in water-tube boilers originally designed for oil-firing. An oil-to-coal ratio of ca. 2 in our final product is within the acceptable ratio range of 1 to 4 required for a typical COM produced by blending finely pulverized coal with heavy fuel oil and various additives (Schumacher, 1981). It is well documented that the combustion of COM containing 13% coal at 20 to 25% ash could yield a boiler efficiency identical to those achieved by burning No. 6 fuel oil, as demonstrated in a New Brunswick power station (Morrison, 1979). With the use of a heavy crude, the coal-in-oil mixture produced from coal tailing's stream using our process is well suited for a commercial high-Btu fuel. The process itself can be engineered at minimum capital and operating cost, as a simple thickener could be used as a reactor and separator. The recycle of the process water

in this single stage process presents another attractive feature both from an economic and environmental point of view.

CONCLUSIONS

1. Hydrophobic fine coals are found to be readily extracted into oil phase. The efficiency of coal extraction is governed by the surface hydrophobicity of coal. A minimum surface hydrophobicity with a critical surface tension of 55 mN/m was needed for effective extraction of coal by oil.
2. Hydrophobic extraction is effective for recovery of coals over a wide particle size range (up to 2 mm) as long as they are sufficiently hydrophobic.
3. The addition of flocculant was found to be unfavorable to coal extraction, but necessary to clarify the process water for recycling. To make this process effective, the extraction of coal with moderate hydrophobicity needs to be accomplished prior to the flocculnt and coagulant addition.
4. Flocculnt or coagulant alone was found inadequate to reduce the suspended solid in process water to a limit of 20 ppm for recycling or discharge. The combination of the two, at a much-reduced dosage, was found to be effective to aggregate fine mineral matters and clays, resulting in a process water suitable for recycling.
5. A process integrating three distinct mechanisms, i.e., hydrophobic extraction, electrolyte coagulation and macromolecular flocculation in a single stage for treating coal tailings is achieved :for
   a) recovering marketable clean coals;
   b) clarifying process water for recycling.
6. A combination of three different mechanisms has been proven successful in retrieving fine coals from the tailings and clarifying process water for recycling in a single stage process. The process provides a potential for the production of a marketable clean fuel, such as COM, from coal tailings. Equally important is the mitigation of the environmental problems by reducing the solid waste disposal and recycling process water.

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Applicant or Patentee: **Jaewon Choung; Jozef Szymanski and Zhenghe Xu**
Serial or Patent No.:
Filed or Issued:
For: **Process for Treating Fine Coal Particles**

VERIFIED STATEMENT (DECLARATION) CLAIMING SMALL ENTITY STATUS (37 CFR 1.9 (f) and 1.27 (b)) - INDEPENDENT INVENTOR

As a below named inventor, I hereby declare that I qualify as an independent inventor as defined in 37 CFR 1.9 (c) for purposes of paying reduced fees under section 41 (a) and (b) of Title 35, United States Code to the Patent and Trademark Office with regard to the invention entitled:

Process for Treating Fine Coal Particles

described in

[X] the specification filed herewith
[ ] Application Serial No.
[ ] Patent no. issued

I have not assigned, granted, conveyed or licensed and am under not obligation, under contract or law to assign, grant, convey or license, any rights in the invention to any person who could not be classified as an independent inventor under 37 CFR 1.9 (c) if that person made an invention or to any concern which would not qualify as a small business concern under 37 CFR 1.9 (d) or a nonprofit organization under 37 CFR 1.9 (e).

Each person, concern or organization to which I have assigned, granted, conveyed or licensed or am under an obligation under contract or law to assign, grant, convey or license any right in the invention is listed below:

[ ] no such person , concern or organization.
[X] persons, concerns or organizations listed below

FULL NAME **Governors of the University of Alberta**

ADDRESS **Suite 222, 8625 - 112 Street, Edmonton, Alberta, Canada T6G 2E1**

I acknowledge the duty to file, in this application or patent, notification of any change in status resulting in loss of entitlement to small entity status prior to paying, or at the time of paying, the earliest of the issue fee or any maintenance fee due after the date on which status as a small entity is no longer appropriate. (37 CFR 1.28 (b)). I hereby declare that all the statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements are made with

the knowledge that wilful false statements and the like so made are punishable by fine or imprisonment, or both under section 1001 of Title 18 of the United States Code, and that such wilful false statements may jeopardize the validity of the application, any patent issuing thereon, or any patent to which the verified statement is directed.

Jaewon Choung
NAME OF INVENTOR

_Signature of Inventor — 5/7/99 Date

Jozef Szymanski
NAME OF INVENTOR

Signature of Inventor — 3.07.99 Date

Zhenghe Xu
NAME OF INVENTOR

_Signature of Inventor — 3-07-99 Date

***NOTE: Separate verified statements are required for each named person, concern or organization having rights to the invention averring to their status as small entities. (37 CFR 1.27)**

Applicant or Patentee: **Jaewon Choung; Jozef Szymanski and Zhenghe Xu**
Serial or Patent No.:
Filed or Issued:
For: **Process for Treating Fine Coal Particles**

VERIFIED STATEMENT (DECLARATION) CLAIMING SMALL ENTITY STATUS (37 CFR 1.9 (f) and 1.27 (D)) - NONPROFIT ORGANIZATION

I hereby declare that I am an official empowered to act on behalf of the nonprofit organization identified below

NAME OF ORGANIZATION **Governors of the University of Alberta**
ADDRESS OF ORGANIZATION **Suite 222, 8625 112 Street, Edmonton Alberta Canada T6G 2E1**

TYPE OF ORGANIZATION [X] University or other institute of higher education

I hereby declare that the nonprofit organization identified above qualifies as a nonprofit organization as defined in 37 CFR 1.9 (e) for purposes of paying reduced fees under section 41 (a) and (b) of Title 35, United States Code with regard to the invention entitled **Process for Treating Fine Coal Particles** inventors **Jaewon Choung; Jozef Szymanski and Zhenghe Xu** described in

[X] the specification filed herewith
[ ] application serial no. , filed

I hereby declare that the rights under contract or law have been conveyed to and remain with the nonprofit organization with regard to the above identified invention.

I acknowledge the duty to file, in this application or patent, notification of any Chuange in status resulting in loss of entitlement to small entity status prior to paying, or at the time of paying, the earliest of the issue fee or any maintenance fee due after the date on which status as a small entity is no longer appropriate. (37 CFR 1.28 (b)).

I hereby declare that all the statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements are made with the knowledge that wilful false statements and the like so made are punishable by fine or imprisonment, or both under section 1001 of Title 18 of the United States Code, and that such wilful false statements may jeopardize the validity of the application, any patent issuing thereon, or any patent to which the verified statement is directed.

NAME OF PERSON SIGNING

Title of Person signing PETER ROBERTSON, Ph.D, P.Eng.
Assoc. Vice President (Research/Industry Relations)
Director, Industry Liaison Office
The University of Alberta

Address of Person Signing

Signature Date July 5th 1999.

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